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(54) Improved perfume carriage and delivery system for laundry applications

Verbessertes Träger- und Freisetzungssystem von Riechstoffen für die Wäschebehandlung

Système amélioré de support et de délivrance de parfums pour le lavage du linge

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• Demeyere, Hugo Jean Marie
B-1881 Merchtem (BE)

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(74) Representative: Canonici, Jean-Jacques et al
Procter & Gamble European Technical Center
N.V.

(73) Proprietor: **THE PROCTER & GAMBLE COMPANY**
Cincinnati, Ohio 45202 (US)

Temseleaan 100
B-1853 Strombeek-Bever (BE)

(72) Inventors:
• Moyaert, Ariane Madeleine Maria
B-8891 Kanegem-Tielt (BE)

(56) References cited:
EP-A- 0 346 034 **FR-A- 2 333 041**
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DescriptionTechnical Field

5 The present invention relates to the general field of laundering. More specifically, this invention relates to the delivery of perfume to fabrics which are washed or treated.

Background

10 The voluminous prior art relating to detergency in general and perfume delivery in particular shows that numerous attempts have been made to design perfume carrier systems which would allow delivering the perfume to the fabrics; in these attempts, the main goal was to achieve delivery of undiluted, undissipated and unaltered perfume to the fabrics, so that the perfume would be released at the end of or after the laundry process.

15 One method for achieving this goal is putting the perfume into a product which goes directly into the dryer. This way, the perfume is delivered to the fabric in the dryer cycle. Such a method is taught in both U.S. Patent 4,511,495, Melville, issued April 16, 1985, and U.S. Patent 4,636,330, Melville, issued January 13, 1987. Both teach forming perfume into particles with a carrier. These particles are then formulated into a composition which is applied to textiles prior to putting into the dryer or prior to clothes-line drying.

20 An even more desirable method for delivering perfume of laundered fabric would be one which provides for protection of the perfume through the washing process and hence delivery of the perfume to fabric in essentially its original state.

25 Such a method must allow for prevention of dilution, degradation or loss of the perfume during the wash cycle of the laundry process. This is done by utilizing a system that releases the perfume in the drying process or later after the perfume has been delivered to the fabric. Preventing release of perfume during the washing process involves very different and more complex technology.

30 One method which has been developed to provide these benefits is perfume microencapsulation. Here the perfume comprises a capsule core which is coated completely with a material which may be polymeric. U.S. Patent 4,145,184, Brain et al, issued March 20, 1979, and U.S. Patent 4,234,627, Schilling, issued November 18, 1980, teach using a tough coating material which essentially prohibits the diffusions out of the perfume. The perfume is delivered to fabric via the microcapsules and is then released by rupture of the microcapsules such as would occur with manipulation of the fabric. A problem with this method is that it takes a certain amount of effort to break the microcapsules.

35 Another method involves providing protection of perfume through the wash cycle and release of perfume in the heat-elevated conditions of the dryer. U.S. Patent 4,096,072, Brock et al, issued June 20, 1978, teaches a method for delivering fabric conditioning agents to textiles through the wash and dry cycle via particles containing hydrogenated castor oil and a fatty quaternary ammonium salt. Perfume may be incorporated into these particles.

All the above systems function quite well as far as the protection of the perfume is concerned; However, these systems do not achieve very well the delivery of perfume to fabrics because quite an amount of the microcapsules is wasted or lost during the wash process, for instance during the rinses.

40 U.S. Patent 4,152,272, Young, issued May 1, 1979, partially addresses this problem in disclosing a perfume delivery system whereby delivery of perfume to the fabrics is said to be enhanced. This patent teaches incorporating perfume into wax particles to protect the perfume through storage in dry compositions and enhance the deposition of the particles on the fabrics during the rinse by the concomitant use of a cationic surfactant. The perfume then diffuses through the wax matrix of the particles on the fabric in the heat-elevated conditions of the dryer.

45 This system is however limited in that it is said to be designed for use in a rinse mode only, i.e. it is not suitable for use in a through the wash mode.

It is an object of the present invention to provide a perfume carriage and delivery system which is suitable for use in a "through the wash" mode, be it in a detergent composition or a fabric treatment composition, or as a laundry additive. It is therefore an object of the present invention to provide a perfume carriage and delivery system which will protect the perfume before and during the wash, and which better deposits on fabrics.

50 Another object of the invention is to provide a system which will gradually release the perfume over a long period of time, after deposition on the fabrics and after the fabrics are dry.

Summary of the Invention

55 It has now been found that the above objects are met when a perfume carriage and delivery system is used which comprises

(a) - from 20% to 80% of perfume;

- (b) - from 5% to 50% of a hydrophobic solid having a melting point of from 30°C to 120°C;
- (c) - from 5% to 50% of C₈₋₂₀ branched or linear alcohol or esters thereof with C₂₋₄ carboxylic acids.
- (d) - from 5% to 50% of an amphiphile polymer.

5 The system will protect the perfume and deliver it to fabrics when it is used in a wash liquor which comprises clay.

Detailed description

The perfume

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Any perfume can be used according to the present invention; it can be relatively simple in its composition or can contain complex mixtures of natural and synthetic chemical compounds which are all chosen to provide any desired odor. Therefore, in its broadest expression, a perfume suitable for use herein is any material which exudes a pleasant or otherwise desirable odor when it is applied to fabrics. Some perfumes which are suitable for use in detergents or fabric treatment compositions are commercially available. Others are described for instance in S. Arctander, Perfume Flavors and Chemicals, Vols. I and II, Author, Montclair, N.J. and the Merck Index, 8th Edition, Merck & Co., Inc. Rahway N.J.

15

According to the present invention, the perfume will represent from 20% to 80% by weight of the total perfume carriage and delivery system (hereinafter referred to as the system). Preferably, the system contains from 40% to 60% by weight of the system of perfume, preferably 50%.

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The hydrophobic solid compound

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The system according to the present invention also comprises a hydrophobic solid compound which has a melting point of from about 30°C to about 120°C. The melting point of the hydrophobic compound influences the melting point of the system, and the melting point of the hydrophobic solid is to be adapted so that the system is solid at ambient temperature but liquid or fluid at wash temperature. Accordingly, the man skilled in the art will need to adapt this parameter according to the climate and washing habits in a given country.

30

By hydrophobic, it is meant that the compound is insoluble in water but miscible with the perfume defined hereinabove. This way, the compound will "protect" the perfume by retaining it in a hydrophobic environment.

35

During the wash, the system will be in a fluid state, due to the warm temperature of the wash water. The system will then form an emulsion in the wash water and deposit on the fabrics, as explained hereinafter. When the rinse occurs, the hydrophobic compound, which still retains the perfume, will "freeze on the fabrics" because of the cold water which is used for rinsing; this way, a very thin layer of the system will be entangled in the fibres of the fabrics, which will not be removed by subsequent rinses.

Hereafter, the perfume will be slowly released from the fabrics by simple diffusion through the hydrophobic compound; this diffusion will be more or less rapid depending on the proportion of hydrophobic solid to perfume. The system can also be designed so as to release perfume from the fabrics even long after they have been washed or treated.

40

The amount of the hydrophobic solid can therefore be adapted by the man skilled in the art, keeping in mind that the higher the amount of hydrophobic solid in the system, the more the perfume is protected, but the less the system disperses in the wash liquor. When a high proportion of hydrophobic solid is used, a slow and durable release of the perfume is obtained. The amount of this hydrophobic solid compound in a system according to the present invention is from about 5% to 50% by weight of the system. Preferred amounts are from 10% to 30% by weight of the system, preferably 15%.

45

Suitable compounds according to the above definition include waxy materials and fats. Such materials may be from mineral, vegetal, animal or synthetic origin. Example include waxes from the petrochemical industry, polyethylene waxes, bees wax, vegetal wax, triglycerides and the like. Highly preferred are mineral waxes from the petrochemical industry.

50

The C₈-C₂₀ alcohols or esters thereof with C₂-C₄ carboxylic acids

The system according to the invention also comprises a C₈-C₂₀ alcohol, or an ester thereof with a C₂-C₄ carboxylic acid, or mixtures thereof.

Preferred compounds are tallow alcohol or myristyl alcohol.

55

This compound is incorporated in the system so as to allow it to better disperse in the wash water. Indeed, it has been observed that the incorporation of this compound in appropriate amounts allows the system to form an emulsion in the wash water upon agitation.

The higher the amount of this compound in the system, the better the dispersion of the system in the wash water,

but more perfume is released in the wash water. In contrast, the lesser the amount of said compound in the system, the poorer the dispersion of the system in the wash water and less perfume is released in the wash water.

The above balance also depends on the amount of hydrophobic material (defined hereinabove) which is incorporated. Indeed, the hydrophobic material tends to protect the perfume but also tends not to disperse very well. It has been observed that the C_8 - C_{20} alcohol or ester thereof with C_2 - C_4 carboxylic acid or mixtures thereof, are to be incorporated in amounts of from 5% to 50% by weight of the total system, preferably from 5% to 25% by weight, preferably 10%.

The amphiphile polymer

With the three types of ingredients defined hereinabove, a system is obtained which protects the perfume and disperses in the wash water. A fourth essential component is an amphiphile polymer which will promote the deposition of the dispersed system on the fabrics during the wash, provided that the system is used in a wash liquor which contains clay.

Suitable polymers for such a purpose are amphiphile polymers derived from such monomers as ethylene oxide, terephthalate propylene oxide, acrylamide, acrylic acid and other polymers.

Particularly preferred are polyethylene oxide/polypropylene oxide block copolymers commercially available under the trade name PLURONIC (R) from BASF.

These polymers are incorporated herein in amounts from 5% to 50% by weight of the total system, preferably from 15% to 35% by weight, preferably 25%.

Industrial application

The perfume carriage and delivery system according to the present invention can be made by mixing together the required ingredients; It is preferred to mix the hydrophobic solid together with the C_8 - C_{20} alcohol or ester thereof with a C_2 - C_4 carboxylic acid, at a temperature above the melting point of the hydrophobic solid; the amphiphile polymer is then added, preferably at such a temperature.

Since most perfumes are quite heat sensitive, it is preferred to allow the above mixture to cool down as much as possible before the addition of the perfume.

Once these steps are completed, the mixture can be left to completely cool down as such, so as to form a block; in a preferred embodiment however, the mixture will be pelletized.

The perfume carriage and delivery system according to the present invention can be used in many different ways; It can be used as such as a laundry additive, for laundry detergent or other fabric treatment compositions which comprise clay; Indeed, as mentioned hereinabove, the presence of clay in the wash liquor is key to a satisfying performance of the system according to the present invention. Clays are conventionally used in the field of detergency, mainly for the purpose of providing softening benefits to fabrics; such clays typically are hectorites or montmorillonites and the like.

It can also be used in a granular detergent composition or a fabric treatment composition comprising clay, i.e. not as an additive anymore, but as an ingredient of a composition.

To allow more flexibility for the user, i.e. to allow the user to use any detergent or fabric treatment composition, including those which do not comprise clay, it is a preferred embodiment of the invention to coagglomerate the system with clay. This way, the system carries its "own" clay, and does not require any other clay supply from the detergent or fabric treatment compositions.

This agglomeration can be made by any agglomerating technique, and these are well known to the man skilled in the art.

In a highly preferred embodiment, the system according to the present invention will not only be agglomerated with clay, but also with a clay-flocculating polymer which will enhance the performance of the system. Suitable clay flocculating polymers are well known to the man skilled in the art, and are described for instance in EP-A-0 299 575, EP 0 313 146 and EP 0 328 182.

The invention is illustrated by the following examples.

EXAMPLES

Example 1

A system according to the present invention is prepared by mixing together

500 g of perfume
150 g of wax ex-TOTAL, cir 40-60; melting point 60°C

100 g of Tallow alcohol
 250 g of polyethylene oxide/polypropylene oxide polymer (pluronic (R) PE 6800 from BASF)

The wax is heated to a temperature of about 70°C, and the tallow alcohol is added while stirring; the polymer is then added, also while stirring. The mixture is then left to cool to a temperature of about 50°C, and the perfume is added. The mixture is then pelletized, and pellets of a system according to the invention are obtained which comprise

50% perfume
 15% wax
 10% Tallow alcohol
 25% polymer

Example 2

A laundry additive comprising a system according to the present invention is obtained by mixing 635 g of clay (High CEC Ca Montmorillonite) with 10 g of polyethylene oxide MW 300 000 (clay-flocculating polymer) in an EIRICH mixer. 200 g of the system of example 1 are then added under high shear mixing, and 155 ml of water are subsequently added. A coagglomerate is then obtained comprising a system according to example 1.

Example 3

A granular detergent composition is made which comprises

Ingredient	Composition (% by weight)
C ₁₁₋₁₂ alkyl benzene sulfonate (Na)	6.5
Tallow alcohol sulfate (Na)	1.0

Ingredient	Composition (% by weight)
Tallow alcohol ethoxylate (EO ₁₁)	0.8
Hydrogenated Tallow fatty acid	1.0
Dodecyl trimethyl ammonium chloride	1.5
Distearyl methyl amine	3.0
Dodecyl dimethyl ammonium N-Oxide	0.4
Zeolite	20.0
Polyethylene oxide (MW=5MM)	0.05
Sodium nitrilotriacetate	5.0
Sodium sulfate	12.7
Sodium silicate	2.0
Sodium perborate (4aq.)	18.0
Carboxymethylcellulose	0.4
Polyacrylate (mw 4000-5000)	3.0
Enzymes (protease, amylase, cellulase)	0.3
Optical brightener	0.25
Sulphonated zinc phthalocyanine	25ppm
EDTA	0.2
Ethylenediamine tetramethylene phosphonic acid	0.1
Tetraacetyl ethylenediamine	1.5
Silicone/silica suds suppressor	0.2
Montmorillonite clay	10.0
System of example 1	0.50

Claims

1. A perfume carriage and delivery system which is suitable for use in a wash liquor comprising clay, characterized in that it comprises a mixture of

- (a) - from 20% to 80% of perfume;
- (b) - from 5% to 50% of a hydrophobic solid having a melting point of from 30°C to 120°C;
- (c) - from 5% to 50% of C₈₋₂₀ branched or linear alcohol or esters thereof with C₂₋₄ carboxylic acids;
- (d) - from 5% to 50% of an amphiphile polymer.

2. A perfume carriage and delivery system according to claim 1, comprising

- from 40% to 60% by weight of the system of component (a)
- from 10% to 30% by weight of the system of component (b)
- from 5% to 25% by weight of the system of component (c)
- from 15% to 35% by weight of the system of component (d).

3. A perfume carriage and delivery system according to claims 1 and 2 comprising:

- 50% of component (a);
- 15% of component (b);
- 10% of component (c);
- 25% of component (d).

4. A perfume carriage and delivery system according to the preceding claims wherein component b is a wax of the petrochemical industry.

5. A perfume carriage and delivery system according to the preceding claims wherein component c is tallow alcohol.

6. A perfume carriage and delivery system according to the preceding claims wherein component d is selected from a polyacrylate, polystyrenesulphonate, polyacrylamide polyethyleneoxide and Block copolymers thereof with polyoxypropylene, polyethylene or polyamide.

7. A perfume carriage and delivery system according to claim 6 wherein said polymer is a Block copolymer of polyethylene oxide and polypropylene oxide.

8. A laundry additive which comprises a perfume carriage and delivery system according to claims 1 thru 7, and clay.

9. A laundry additive according to claim 8 wherein the system and the clay are coagglomerated.

10. A laundry additive according to claims 8 and 9 which additionally comprises a clay flocculating polymer.

11. A granular detergent composition comprising conventional detergent ingredients, clay, and a perfume carriage and delivery system according to claims 1 thru 7.

12. A through the wash softening composition comprising clay and a perfume carriage and delivery system according to claims 1 thru 7.

Patentansprüche

1. Träger- und Freisetzungssystem für Parfum, das für die Verwendung in einer Waschlauge geeignet ist und Ton enthält, dadurch gekennzeichnet, daß es ein Gemisch aus

- (a) - 20% bis 80% Parfum;
- (b) - 5% bis 50% von einem hydrophoben Feststoff mit einem Schmelzpunkt von 30°C bis 120°C;
- (c) - 5% bis 50% von einem verzweigten oder linearen C₈₋₂₀-Alkohol oder von Estern hiervon mit C₂₋₄-Carbonsäuren;

(d) - 5% bis 50% von einem amphiphilen Polymer umfaßt.

2. Träger- und Freisetzungssystem für Parfum nach Anspruch 1, umfassend

- 5
- 40% bis 60%, bezogen auf das Gewicht des Systems, von der Komponente (a);
 - 10% bis 30%, bezogen auf das Gewicht des Systems, von der Komponente (b);
 - 5% bis 25%, bezogen auf das Gewicht des Systems, von der Komponente (c);
 - 15% bis 35%, bezogen auf das Gewicht des Systems, von der Komponente (d).

10 3. Träger- und Freisetzungssystem für Parfum nach den Ansprüchen 1 und 2, umfassend

- 15
- 50% von der Komponente (a);
 - 15% von der Komponente (b);
 - 10% von der Komponente (c);
 - 25% von der Komponente (d).

4. Träger- und Freisetzungssystem für Parfum nach den vorstehenden Ansprüchen, wobei die Komponente b ein Wachs aus der petrochemischen Industrie ist.

20 5. Träger- und Freisetzungssystem für Parfum nach den vorstehenden Ansprüchen, wobei die Komponente c Talgalkohol ist.

25 6. Träger- und Freisetzungssystem für Parfum nach den vorstehenden Ansprüchen, wobei die Komponente d unter Polyacrylat, Polystyrolsulfonat, Polyacrylamid, Polyethylenoxid und Blockcopolymeren hievon mit Polyoxypropylen, Polyethylen oder Polyamid ausgewählt ist.

7. Träger- und Freisetzungssystem für Parfum nach Anspruch 6, wobei das genannte Polymer ein Blockcopolymer aus Polyethylenoxid und Polypropylenoxid ist.

30 8. Zusatzmittel zum Wäschewaschen, welches ein Träger- und Freisetzungssystem für Parfum nach den Ansprüchen 1 bis 7 und Ton umfaßt.

9. Zusatzmittel zum Wäschewaschen nach Anspruch 8, wobei das System und der Ton gemeinsam agglomeriert sind.

35 10. Zusatzmittel zum Wäschewaschen nach den Ansprüchen 8 und 9, welches zusätzlich ein Tonflockungspolymer umfaßt.

40 11. Körnige Detergenzzusammensetzung, umfassend herkömmliche detergierende Bestandteile, Ton und ein Träger- und Freisetzungssystem für Parfum nach den Ansprüchen 1 bis 7.

12. Zusammensetzung zum Weichmachen während des Waschens, umfassend Ton und ein Träger- und Freisetzungssystem für Parfum nach den Ansprüchen 1 bis 7.

45 **Revendications**

1. Un système de support et pour délivrer un parfum qui est approprié pour être utilisé dans une liqueur de lavage comprenant de l'argile, caractérisé en ce qu'il comprend un mélange de :

- 50
- (a) 20% à 80% de parfum ;
 - (b) 5% à 50% d'un solide hydrophobe ayant un point de fusion de 30°C à 120°C ;
 - (c) 5% à 50% d'un alcool en C₈₋₂₀, ramifié ou linéaire ou ses esters avec des acides carboxyliques en C₂₋₄ ;
 - (d) 5% à 50% d'un polymère amphiphile.

55 2. Un système de support et pour délivrer un parfum selon la revendication 1, comprenant :

- de 40% à 60% en poids du système du composant (a)
- de 10% à 30% en poids du système du composant (b)

- de 5% à 25% en poids du système du composant (c)
- de 15% à 35% en poids du système du composant (d).

3. Un système de support et pour délivrer un parfum selon les revendications 1 et 2 comprenant :

- 50% du composant (a) ;
- 15% du composant (b) ;
- 10% du composant (c) ;
- 25% du composant (d) ;

4. Un système de support et pour délivrer un parfum selon les revendications précédentes, dans lequel le composant b est une cire de l'industrie pétrochimique.

5. Un système de support et pour délivrer un parfum selon les revendications précédentes, dans lequel le composant c est de l'alcool de suif.

6. Un système de support et pour délivrer un parfum selon les revendications précédentes, dans lequel le composant d est choisi parmi un polyacrylate, du sulfonate de polystyrène, du polyacrylamide, de l'oxyde de polyéthylène et leurs copolymères séquencés avec du polyoxypropylène, du polyéthylène ou du polyamide.

7. Un système de support et pour délivrer un parfum selon la revendication 6, dans lequel ledit polymère est un copolymère séquencé d'oxyde de polyéthylène et d'oxyde de polypropylène.

8. Un additif de blanchiment qui comprend un système de support et pour délivrer un parfum selon les revendications 1 à 7 et de l'argile.

9. Un additif de blanchiment selon la revendication 8, dans lequel le système et l'argile sont co-agglomérés.

10. Un additif de blanchiment selon les revendications 8 et 9 qui comprend supplémentairement un polymère floculant l'argile.

11. Une composition détergente granulaire comprenant des ingrédients détersifs classiques, de l'argile et un système de support et pour délivrer un parfum selon les revendications 1 à 7.

12. Une composition d'adoucissant de lavage comprenant de l'argile et un système de support et pour délivrer un parfum selon les revendications 1 à 7.